

CHEMICAL CHARACTERIZATION OF THE PYROTECHNICALLY DISSEMINATED XM30 MAIN GUN SIGNATURE SIMULATOR

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ABSTRACT

Development of the Multiple Integrated Laser Engagement System (MILES) 2000 device has been selected as the replacement to the currently used Hoffman systems. On the M1 or M60 tanks, a laser is activated to simulate firing of the main gun, which triggers a signal to the Main Gun Signature Simulator (MGSS) to activate the XM30 simulator. The XM30 simulator was designed to produce a flash and bang so the tank crew will receive a visual as well as an audible confirmation of the firing.

The purpose of this study was to pyrotechnically disseminate the XM30 simulators in a controlled chamber environment and to maintain a specified concentration range for chemical characterization of the combustion products. Data was needed prior to material release and for incorporation into the item's Life Cycle Environmental Assessment (LCEA). Characterization was to principally include compounds of toxicological significance such as benzene, formaldehyde and carbon monoxide, but detection of other volatile organic compounds, inorganic metals, and particle size analysis were also performed. The inorganic gases NO_x and SO_x were not analyzed for because previous data had already shown their concentrations to be far below their TLV-TWA's. A detailed mass balance was also well outside the scope of this study.

For it's current use in combat training exercises, this study provides evidence that the combustion products produced by firing of the XM30 simulator are of a safe and non-toxic nature.

1. INTRODUCTION

Development of the Multiple Integrated Laser Engagement System (MILES) 2000 device has been selected as the replacement to the currently used Hoffman systems. These devices are used in battlefield training exercises to simulate the flash / bang firing of a battle tank. On the M1 or M60 tanks, a laser is activated to simulate firing of the main gun, which triggers a signal to the Main Gun Signature Simulator (MGSS) to activate the XM30 simulator. The XM30 simulator was designed to produce a flash and bang so the tank crew will receive a visual as well as an audible confirmation of the firing.¹

Report Documentation Page				Form Approved OMB No. 0704-0188	
Public reporting burden for the collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to a penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.					
1. REPORT DATE 00 JAN 2002		2. REPORT TYPE N/A		3. DATES COVERED -	
4. TITLE AND SUBTITLE Chemical Characterization Of The Pyrotechnically Disseminated Xm30 Main Gun Signature Simulator				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S)				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Edgewood Chemical Biological Center (ECBC) Bldg. E3150, Aberdeen Proving Ground, MD. 21010-5423; Geo-Centers, Inc. Aberdeen Proving Ground , MD. 21010-5423				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release, distribution unlimited					
13. SUPPLEMENTARY NOTES This article is from ADA409494 Proceedings of the 2001 ECBC Scientific Conference on Chemical and Biological Defense Research, 6-8 March , Marriott's Hunt Valley Inn, Hunt Valley, MD., The original document contains color images.					
14. ABSTRACT					
15. SUBJECT TERMS					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UU	18. NUMBER OF PAGES 20	19a. NAME OF RESPONSIBLE PERSON
a. REPORT unclassified	b. ABSTRACT unclassified	c. THIS PAGE unclassified			

Prior to fielding, a detailed Health Hazard Assessment (HHA) and Life Cycle Environmental Assessment (LCEA) were prepared for review.^{2,3,4} Although these documents have been completed, additional information on the chemical characterization of the combustion byproducts was needed. This information is vital to assure that soldiers conducting combat training exercises with the simulators are not being exposed to potentially harmful byproducts.

The purpose of this study was to pyrotechnically disseminate the XM30 simulators in a controlled environment and to maintain a specified concentration range for chemical characterization of the combustion products. The inorganic gases NO_x and SO_x were not sampled during the characterization for two reasons. First, the Health Hazard Assessments had stated that previous detection of them did not exceed standards for exposure inside the M1 hatch.^{2,3} Second, previous studies containing similar starting materials at higher quantities had also shown levels of NO_x and SO_x not to exceed their TLV-TWA's.⁵ Initial characterization was to principally include compounds of toxicological significance, such as benzene, formaldehyde, and carbon monoxide, that have been shown to exist in combustion analyses from other smoke studies. Detection of other volatile organic compounds (VOC's), inorganic metals, and particle size analysis were also performed. A detailed mass balance was not within the scope of work defined by this study.

2. MATERIALS AND METHODS

2.1 MATERIALS - XM30 SIMULATORS

The XM30 is an electric match initiated device consisting of a thermoplastic elastomere cap, polypropylene case, flash/smoke charge, and nickel plated brass contact pins. Figure 1 shows an exterior view of the simulator and figure 2 shows a more detailed interior view. A listing of the chemicals and their respective percentages found in the flash/smoke charge is shown in Table 1. Each of the simulators initially contains about 6 grams of pyrotechnic / energetic material. All of the simulators (Lot # CPA99A004-003) were manufactured by Comet GmbH in Germany and transported from Picatinny Arsenal to the Edgewood Chemical and Biological Center's (ECBC) Engineering Directorate. On the days of testing, a simulator was received and delivered to the pyrotechnic chamber at Bldg. E3266. All test items were weighed before and after dissemination to assure complete combustion of the smoke charge

2.2 CHAMBER EXPOSURE SYSTEM

The simulator was clamped loosely on a metal table in the middle of the 20,000 liter pyrotechnic chamber (figure 3). While wearing a grounding strap, two alligator clips were connected to the contact pins, and run to an electric discharge device (Black Magic, Pyromate Inc.) about 4-5 meters from the outside of the chamber. The door to the chamber was secured and latched, the electric discharge device was activated, and the simulator was fired. As recorded in the interim hazard classification, the sound output of the simulator is 135 decibels at a distance of 20 meters.¹ Audio confirmation of firing from a distance of 4-5 meters was therefore easily detected. Following dissemination, the fan was activated in the chamber to provide uniform dispersion and the smoke was diverted through a 4 inch diameter duct to a smaller 500 liter chamber (figure 4). Chamber environmental parameters monitored during all of the tests were temperature, relative humidity, and airflow.

TABLE 1. Chemical components of the XM30 flash / smoke charge.

Chemical
*Potassium nitrate (CAS # 7757-79-1)
Magnesium (CAS # 7439-95-4)
Black powder
Silica, amorph (CAS # 112945-52-5)
* preprocessed with 3% of boiled linseed oil

2.3 CHAMBER CONCENTRATION

To monitor chamber concentration, 25 mm A/E glass fiber filter pads (Gelman Scientific) were used to collect particulate samples for total aerosol concentration at 5, 15 and 25 minute intervals during a 30-minute test. Forty liters of air (10 l/min x 4 min) from the 500 liter chamber were drawn onto the pads using a vacuum pump (Sierra Instruments). Gravimetric analysis was performed on the resulting pads using a Cahn microbalance to determine chamber concentration.

2.4 PARTICLE SIZE ANALYSIS

The Thermal Systems Incorporated (TSI) Aerodynamic Particle Sizer (APS) system with model 3302 diluter was used to determine the particle size and distribution from the 500 liter chamber. The diluter was necessary to avoid overloading the APS system.⁶ Several backgrounds were run of the 500-liter chamber prior to collecting data at the five, fifteen and twenty-five minute time intervals to assure the absence of any particles. All instrument specifications are provided in Appendix A.

2.5 CHEMICAL CHARACTERIZATION

2.5.1 VOLATILE ORGANIC COMBUSTION PRODUCTS (VOC'S)

At 5, 15, and 25 minutes into the run, VOC's of the combusted smoke were collected from the 500 liter chamber onto tenax tubes for subsequent analysis by thermal desorption gas chromatography/mass spectrometry (Appendix B for instrument conditions). Samples were drawn from two ports of the 500-liter chamber at 200 ml/min for five minutes. Background checks prior to grenade dissemination were performed to rule out potential contaminants and to allow background subtraction by Gas Chromatography/Mass Spectrometry (GC/ MS).

2.5.2 FORMALDEHYDE ANALYSIS

Previous characterization studies of other disseminated smoke materials had shown the presence of formaldehyde (CAS # 50-00-0) after combustion.⁵ LpDNPH air monitoring cartridges (Supelco, Inc.) were used to trap formaldehyde on a high purity silica absorbent coated with 2,4-dinitrophenylhydrazine (CAS # 119-26-6). Over 30 minutes, the maximum flow rate through the tube (2 l / min) was initially used to determine the presence or absence of formaldehyde. Following collection, the samples were eluted with high purity acetonitrile (CAS # 75-05-8), and analyzed using gas chromatography / flame ionization detection (Appendix C for instrument conditions). Standards of 0.91, 2.74, 5.49, 12.80, and

18.29 ug/ml were prepared in acetonitrile from formaldehyde 2,4 -dinitrophenylhydrazone (CAS # 1081-15-8). Under this method, the detection limit for formaldehyde was determined to be 2 ug/ml.

2.5.3 INORGANIC ANALYSIS (SOLID)

Previous environmental assessments had determined that a majority of the solid material collected after dissemination of the XM30 simulators were potassium or magnesium carbonates and hydroxides.⁴ Atomic Absorption (AA) spectroscopy was used to look at elemental potassium (CAS # 7440-09-7) and magnesium (CAS # 7439-95-4) and to quantitatively determine the percentages of all solid combustion products containing either of these elements. Due to the inherent nontoxic nature of these compounds, it was not within the stated objectives to separate the percent magnesium and potassium compounds into their individual carbonate and hydroxide percentages. Past work on other smoke devices with similar materials and higher quantities than those listed in Table 1 have also shown levels of SO_x and NO_x to be far below their respective Threshold limit value – Time Weighted Averages (TLV-TWA's).⁵ Solid material from the 500 liter chamber was collected onto the 25 mm A/E glass fiber filter pads and solvent desorbed. Pads could not be analyzed for potassium and magnesium simultaneously because the method used to dissolve magnesium would reduce the observable response for potassium.⁷

2.5.3.a POTASSIUM ANALYSIS

Since potassium is water soluble⁷, the filter pad was placed in a glass beaker, rinsed with 10 ml of deionized water, and sonicated for approximately 5 minutes. Solid material from the pad was completely desorbed into solution. Sample aliquots dependent on the amount of initial pad material were transferred and diluted into 100 ml of water. Standards of 0.5, 1.0, 5.0 and 10.0 ug/ml were prepared from a 1000 ppm atomic absorption standard (EM Science).

2.5.3.b MAGNESIUM ANALYSIS

Magnesium compounds are relatively insoluble in water, and therefore needed to be dissolved in acid prior to analysis by AA.⁷ Ten ml of concentrated hydrochloric acid was added, heated for a few minutes, transferred, and diluted into 100 ml. Standards of 0.5, 1.0, 5.0 and 10.0 ug/ml were prepared from a 1000 ppm atomic absorption standard (EM Science).

2.5.4 INORGANIC ANALYSIS (GAS)

The AIM electrochemical Logic Series 500/501 gas detector was used in a diffusion mode to monitor carbon monoxide that may have appeared from combustion. Sampling was performed by placing the detector inside the small 500-liter chamber and recording any response seen at the sampling intervals.

3. RESULTS

All statistical analysis were performed using the Jandel computer software package Sigma Stat 2.03 for Windows.

3.1 CHAMBER EXPOSURE SYSTEM

Daily adjustments were made to chamber flow rates due to ambient conditions (humidity, temperature, and pressure differential at the exhaust vent due to outside wind velocity) in an attempt to maintain a predetermined chamber concentration. However, these fluctuations in chamber concentrations become more evident as the target chamber concentration dropped. With a small amount of material

initially contained in the XM30 simulator, these fluctuations were inevitable in the described dynamic system. Therefore, it was assumed that there would be differences between sample days that could not be avoided and that the chemical characterization would be reported over a concentration range.

3.2 CHAMBER CONCENTRATION BETWEEN SAMPLING DAYS

A Two Way ANOVA / Tukey Test was performed to compare the differences between sampling days while eliminating any variance between sampling intervals. As expected, results confirmed that there were chamber concentration differences between sampling days (figure 5). Table 2 also lists the days that were significantly different at $p \leq 0.05$.

3.3 MEAN CHAMBER CONCENTRATION

A Two Way Analysis of Variance (ANOVA) / multiple comparison test (Tukey Test) was also performed to determine if there were differences between sampling intervals (5-9 min, 15-19 min and 25-29 min) while accounting for the variability between sampling days. Results confirmed that there were significant differences between all three sampling intervals at $p \leq 0.05$ (figure 6). The mean chamber concentrations were 182.5 ± 4.9 , 144.7 ± 4.9 , and 115.5 ± 4.9 mg/m³. In a dynamic system, it would be expected that chamber concentrations would fall with time, but the study was designed to obtain the smallest concentration range over which chemical characterization was performed.

3.4 PARTICLE SIZE ANALYSIS

The mass median aerodynamic diameters (MMAD's) of the particles were 1.27 , $\sigma_g = 1.1$, 1.25, $\sigma_g = 1.1$ and 1.21, $\sigma_g = 1.1$ um at the sampling intervals (figure 7). Although the size of the particles is in the respirable range, chemical characterization results will show them to be non-hazardous.

3.5 CHEMICAL CHARACTERIZATION

3.5.1 VOLATILE ORGANIC COMBUSTION PRODUCTS

A small amount of material present in the XM30 simulator caused characterization to be performed on a low mean concentration range of generated smoke in the 500-liter chamber. With a total sample volume of one liter drawn onto a Dynatherm tube, collected combustion products were expected to be small and not deviate much from the inherent background of the chamber. Therefore it was important to collect background tubes each day prior to dissemination (figure 8) and to subtract them from the collected samples. There are some small peaks seen in the background but this is a result of the high sensitivity of the analytical technique (thermal desorption GC/MS). To illustrate this, figure 9 compares the large response of a 0.262 ug spiked benzene standard with the background and equation 1 shows the corresponding concentration.

$$\text{Equation 1: } \text{ppm} = \frac{[(\text{benzene collected (ug)} / \text{total volume drawn (l)})](24.46 \text{ l/mole})}{\text{MW benzene (g/mol)}} = .0821 \text{ ppm}$$

Some of the background-subtracted chromatograms typically seen for the XM30 simulator are shown in figures 10-12. Within the specified concentration range, responses seen on the tenax tubes were either slight deviations above the chamber background or extremely low concentrations of VOC's produced from dissemination. Chromatograms and peak responses will differ slightly between sampling days and at differing sampling times due to the statistical results presented earlier. Slight differences in abundance were also observed between the two ports, but only because responses were recorded near or

at the instrument's detection limit. All detectable VOC's seen and presented in Table 3 were of low response, and those of toxicological significance were far below their TLV-TWA's.⁸ For example, after making a response factor comparison with the 0.262 ug benzene (CAS # 71-43-2) standard, the benzene response seen in figure 10 (retention time 3.58 min) corresponds to an approximate quantity of 1.05×10^{-2} ug. Insertion into equation 1 yields .003 ppm (or 3 ppb), which is approximately 150 times below benzene's TLV-TWA. By observation, quantitation of the other peaks was therefore not necessary due to their similar small responses. There were no peaks that were significantly different from the benzene response to allow them to approach their TLV. The largest three peaks on the chromatograms at retention times 11.13, 15.48, and 21.62 were results of column bleed due to a low column flow rate and slow temperature ramp approaching elevated temperatures. These experimental conditions were necessary for peak resolution and complete detection of all possible VOC's.

3.5.2 FORMALDEHYDE ANALYSIS

Quantitation of formaldehyde (HCHO) concentration was calculated according to equation 2. At a detection limit of 2 ug/ml, minimal elution volume of 2 ml, and maximum air sample of 60 liters, the concentration of formaldehyde was calculated to be 0.054 ppm (approximately 5 times lower than the established TLV-STEL of 0.3 ppm). Since there was no formaldehyde detected at this concentration, any formaldehyde that may be present in the combustion products must be at a level below this detection limit concentration.

$$\text{Equation 2: ppm} = \frac{[(\text{HCHO (ug/ml)})(\text{elution volume (ml)}) / \text{total volume drawn (l)}](24.46/\text{mole})}{(\text{ug/g}) \quad \text{MW formaldehyde (g/mol)}}$$

3.5.3 INORGANIC ANALYSIS (SOLID)

Magnesium and potassium analysis was calculated as a percentage of the total residue collected on the filter pads and recorded as elemental magnesium and potassium compounds (figure 13). Since the results were determined as a percentage of total residue collected, it was not necessary to account for daily variability. A One Way ANOVA was performed to determine the differences between sampling intervals. There were no significant differences between magnesium concentrations or potassium concentrations at the sampling intervals. The mean magnesium compound concentrations were 33.9 ± 3.0 , 32.9 ± 3.7 and 32.1 ± 3.3 % and the mean potassium compound concentrations were 16.3 ± 3.4 , 17.2 ± 1.2 and 17.8 ± 2.3 %.

3.5.4 INORGANIC ANALYSIS (GAS)

The AIM electrochemical Logic Series 500/501 gas detector did not sense the presence of carbon monoxide down to 1 ppm at the 5, 15 and 25-minute sampling intervals. The TLV-TWA for carbon monoxide as established by the ACGIH is 25 ppm.

4. DISCUSSION

Prior to material release of the XM30 simulator, work was required to chemically characterize the combustion products. Additional data was needed for incorporation into the item's Life Cycle Environmental Assessment (LCEA). Characterization was to principally include compounds of toxicological significance such as benzene, formaldehyde and carbon monoxide, but detection of other volatile organic compounds, inorganic metals, and particle size analysis were also performed. The inorganic gases NO_x and SO_x were not analyzed for because previous data had already shown their concentrations to be far below their TLV-TWA's⁵.

With only 6 grams of starting material contained in the pyrotechnic charge, an initial concern was whether the simulator would generate enough smoke for combustion product analysis to be performed. The main issue to overcome was that the simulators had to be disseminated from the large pyrotechnic chamber for safety reasons, but sampling was easier to perform from the smaller chamber. The smoke was therefore diverted from the large chamber and dynamically run for 30 minutes through the small chamber. The mean chamber concentration range at which characterization took place was from 182.5-115.5 mg/m³. This corresponds to a low chamber concentration (approximately 150 mg/m³) at which other documented reports have successfully performed characterizations of alternate smoke materials.⁹ Statistical analysis did reveal chamber concentration differences between sampling days and between sampling intervals (5,15, 25 minutes), but all characterization was performed within the mean chamber concentration range.

Benzene, formaldehyde, and carbon monoxide were at concentration levels far below their respective TLV-TWA's. These results were somewhat expected due to the initially small amount of starting material present in the simulators. Other trace VOC's detected by thermal desorption GC/MS included several different carbonyl derivatives. Aldehydes (but-, hex-, non-, dec-, and benzaldehyde), ketones (acetone and hexanone) and amides (2-propanamide) were all seen. The non-carbonyl compound hexanol was also seen at trace levels.

Following combustion, inorganic analysis showed that nearly 50% of the solid material were compounds containing either magnesium or potassium. Most likely, they would be present as hydroxides or carbonates. At the 5, 15, and 25 minute time intervals, the mean magnesium compound concentrations were 33.9 ± 3.0 , 32.9 ± 3.7 and 32.1 ± 3.3 % and the mean potassium compound concentrations were 16.3 ± 3.4 , 17.2 ± 1.2 and 17.8 ± 2.3 %. Release of these metal compounds into the environment should not cause any environmental impact; however, there have been no studies that examined what long-term effects the salt concentrations might have on aquatic salt-sensitive organisms.

Particle size analysis showed MMAD's of the particles to be 1.27, $\sigma_g = 1.1$, 1.25, $\sigma_g = 1.1$ and 1.21, $\sigma_g = 1.1$ um at the sampling intervals. These particles are considered small and in the respirable range, but quick diffusion of the smoke cloud and it's inherent nontoxic nature have already been shown.

5. CONCLUSIONS

Chemical characterization of pyrotechnically disseminated smoke released from the XM30 main gun signature simulator did not produce toxicologically important compounds at concentration levels above their respective TLV-TWA's. Benzene, formaldehyde, and carbon monoxide were far below their threshold values. Other VOC's detected in trace amounts included various short chain carbonyl compounds and an alcohol compound. Inorganic analysis of the solid residue showed a high percentage of magnesium / potassium compounds, and particle size analysis showed the production of small diameter particles.

For it's current use in combat training exercises, this study provides evidence that the combustion products produced by firing of the XM30 simulator should not place the soldier in hazardous conditions. Data will be used for input into the item's Life Cycle Environmental Assessment (LCEA).

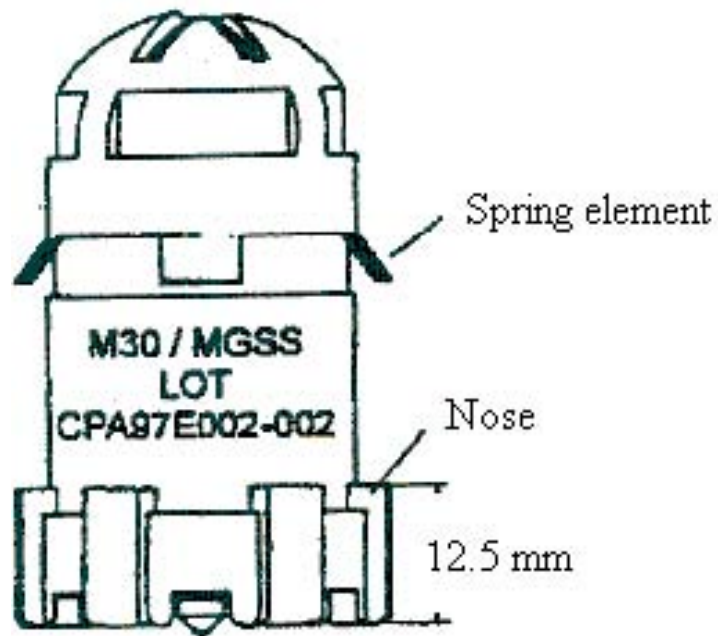


Figure 1. Exterior side view of XM30 simulator.

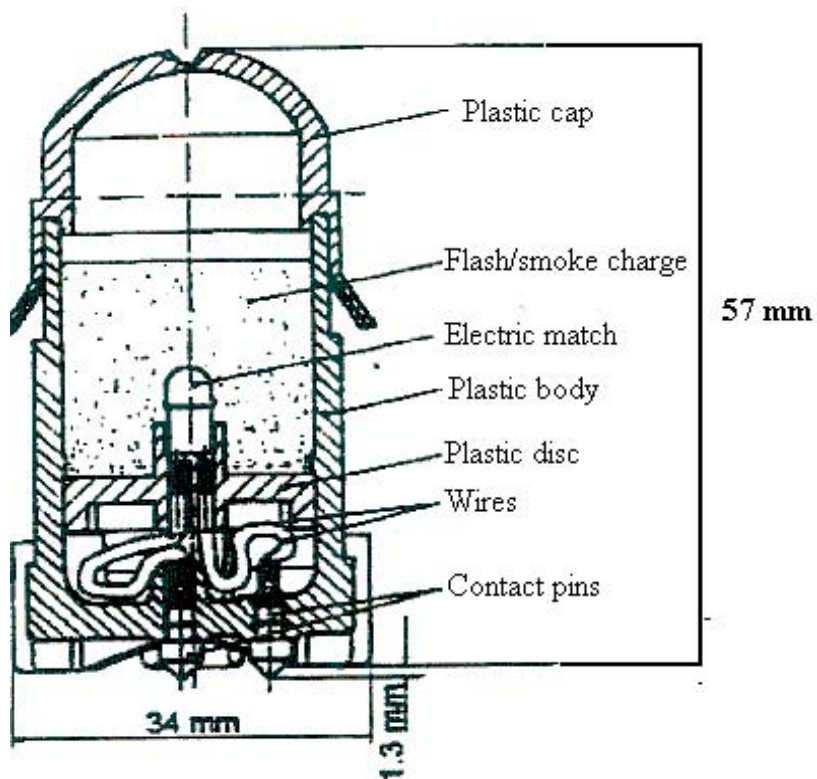


Figure 2. Interior view of XM30 simulator.

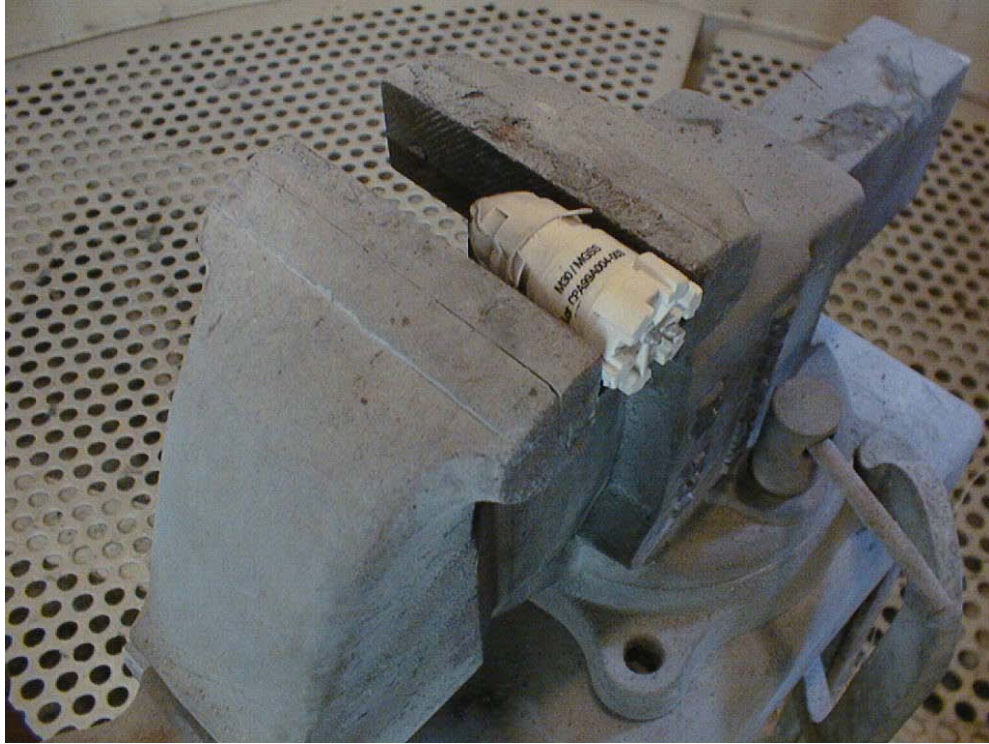


Figure 3. XM30 simulator clamped in 20,000 liter chamber.



Figure 4. 20,000 pyrotechnic chamber diverted to 500 liter sampling chamber.

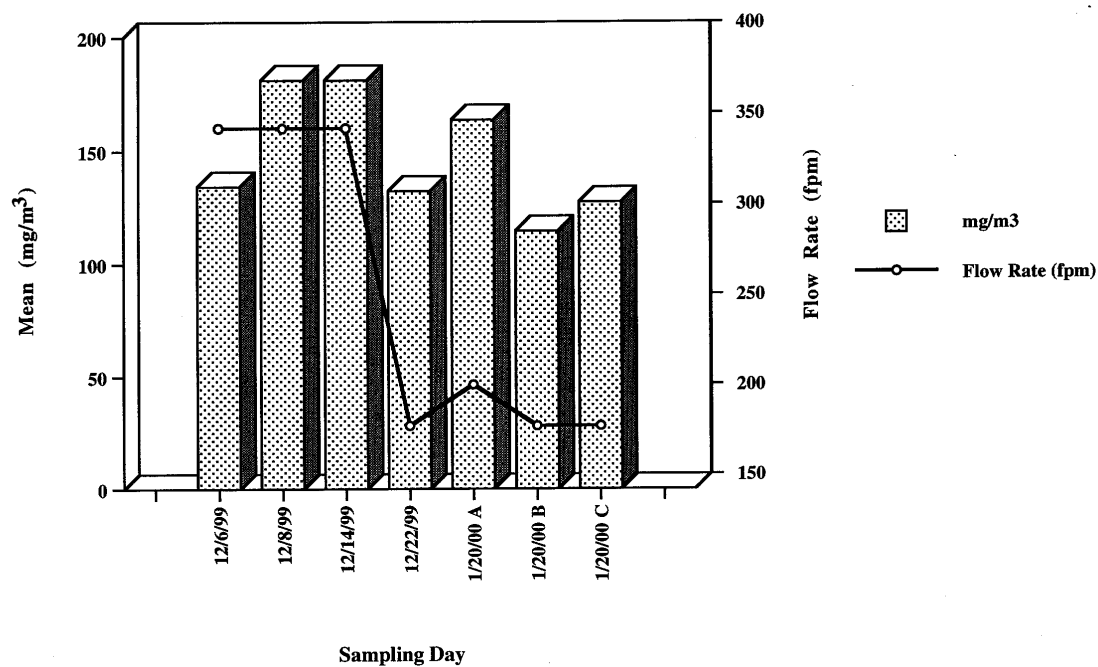


Figure 5. Chamber concentration differences between sampling days.

TABLE 2. Multiple Comparison for Chamber Concentration.

Run Dates	12/6/99	12/8/99	12/14/99	12/22/99	1/20/00 A	1/20/00 B	1/20/00 C
12/6/99							
12/8/99	yes						
12/14/99	yes	no					
12/22/99	no	yes	yes				
1/20/00 A	no	no	no	no			
1/20/00 B	no	yes	yes	no	yes		
1/20/00 C	no	yes	yes	no	no	no	

The multiple comparison test used was the Tukey Test.
 $P \leq 0.05$, Power of performed test was 1.0.

Yes - significant difference between sampling days.
 No - no significant difference between sampling days.

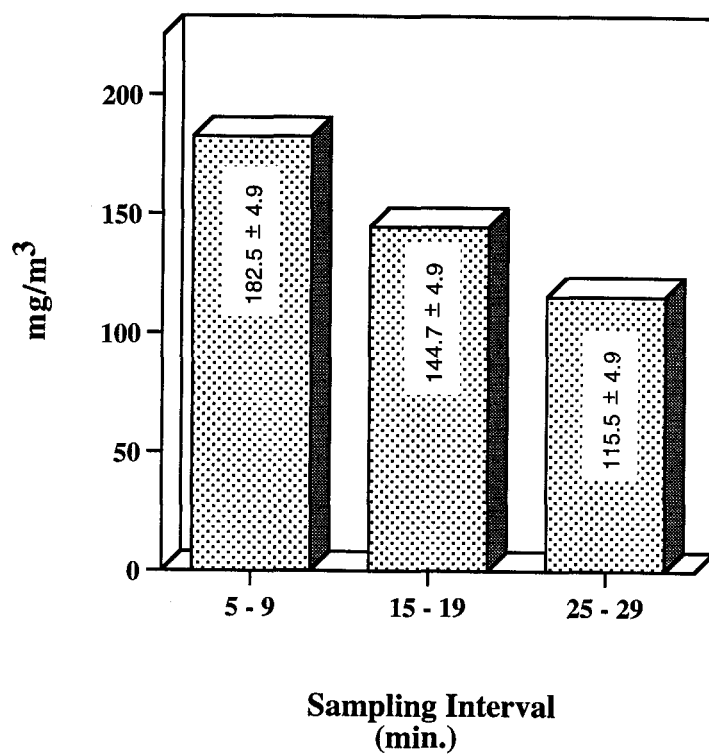


Figure 6. Mean chamber concentrations at sampling intervals.

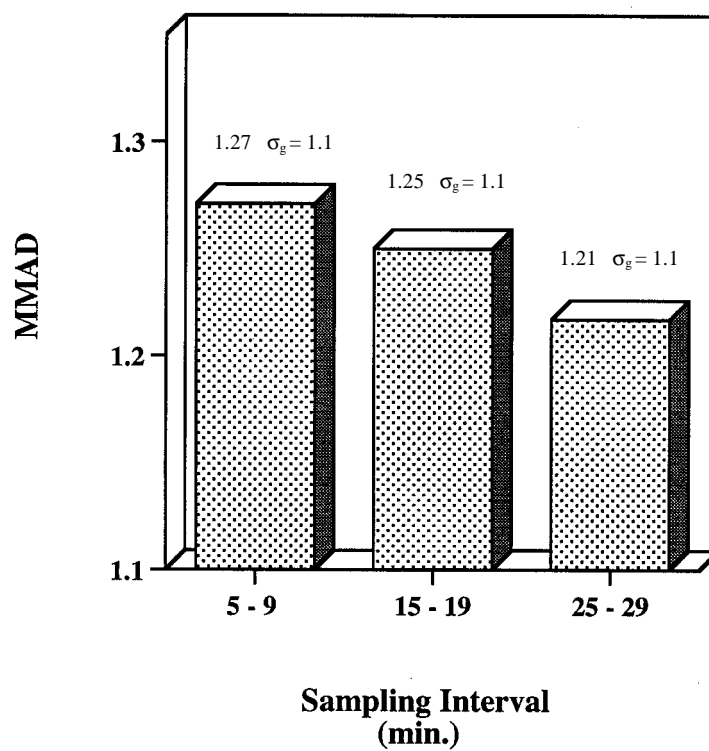


Figure 7. Mean particle size at sample intervals.

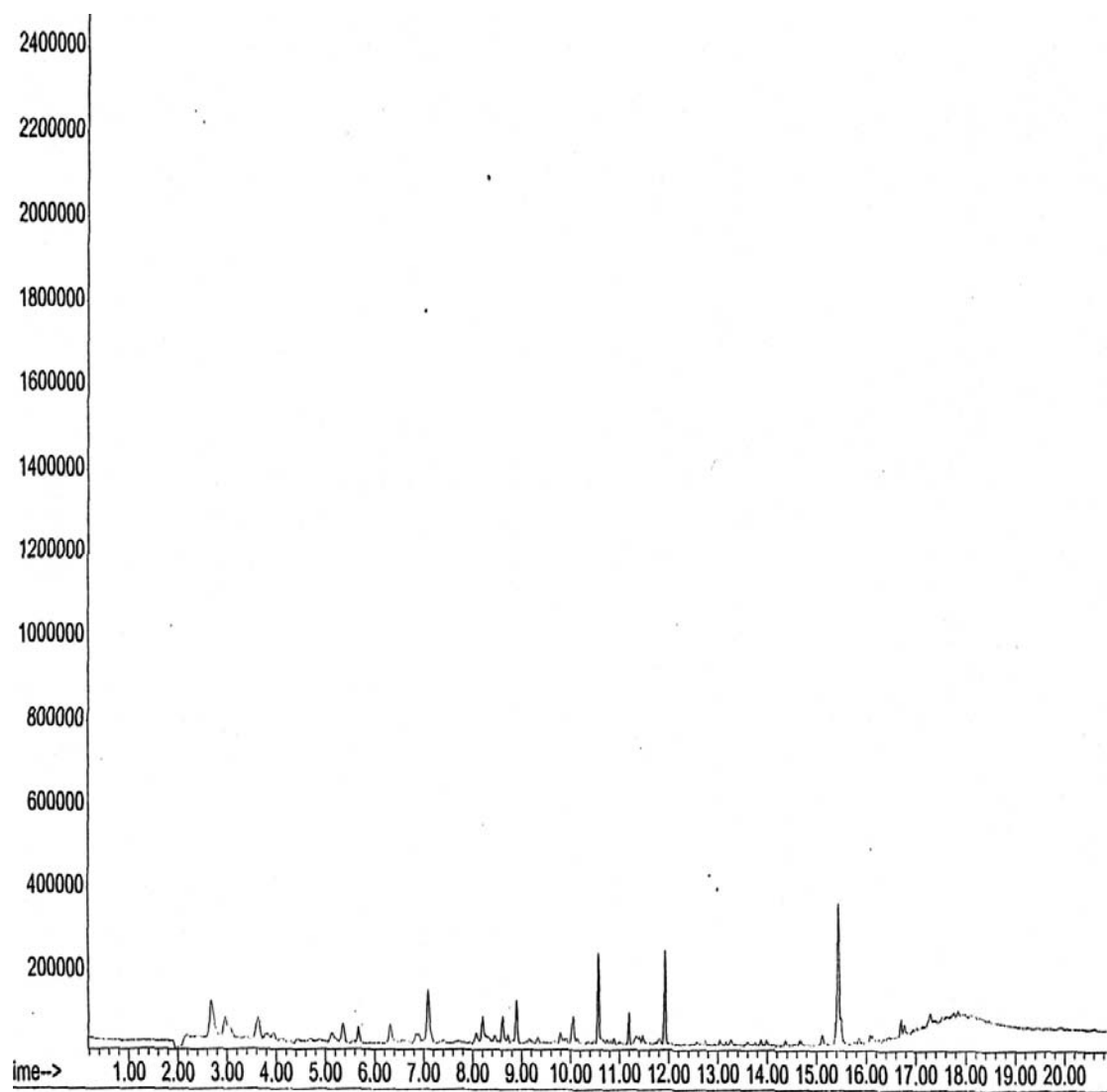


Figure 8. Sample background chromatogram from 500 liter chamber.

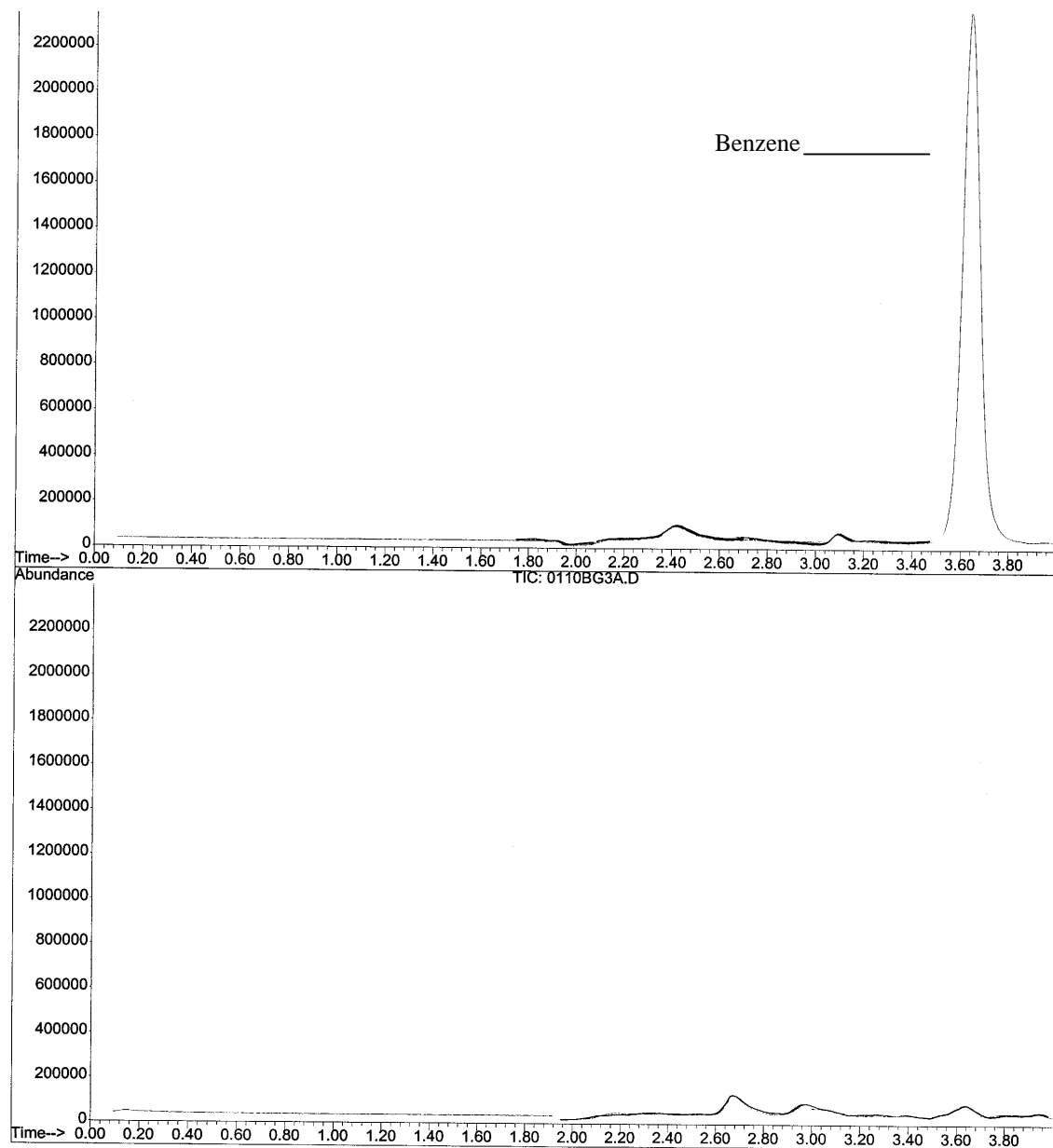


Figure 9. Comparison of 0.262 ug spiked benzene peak against chamber background (0.0821 ppm).

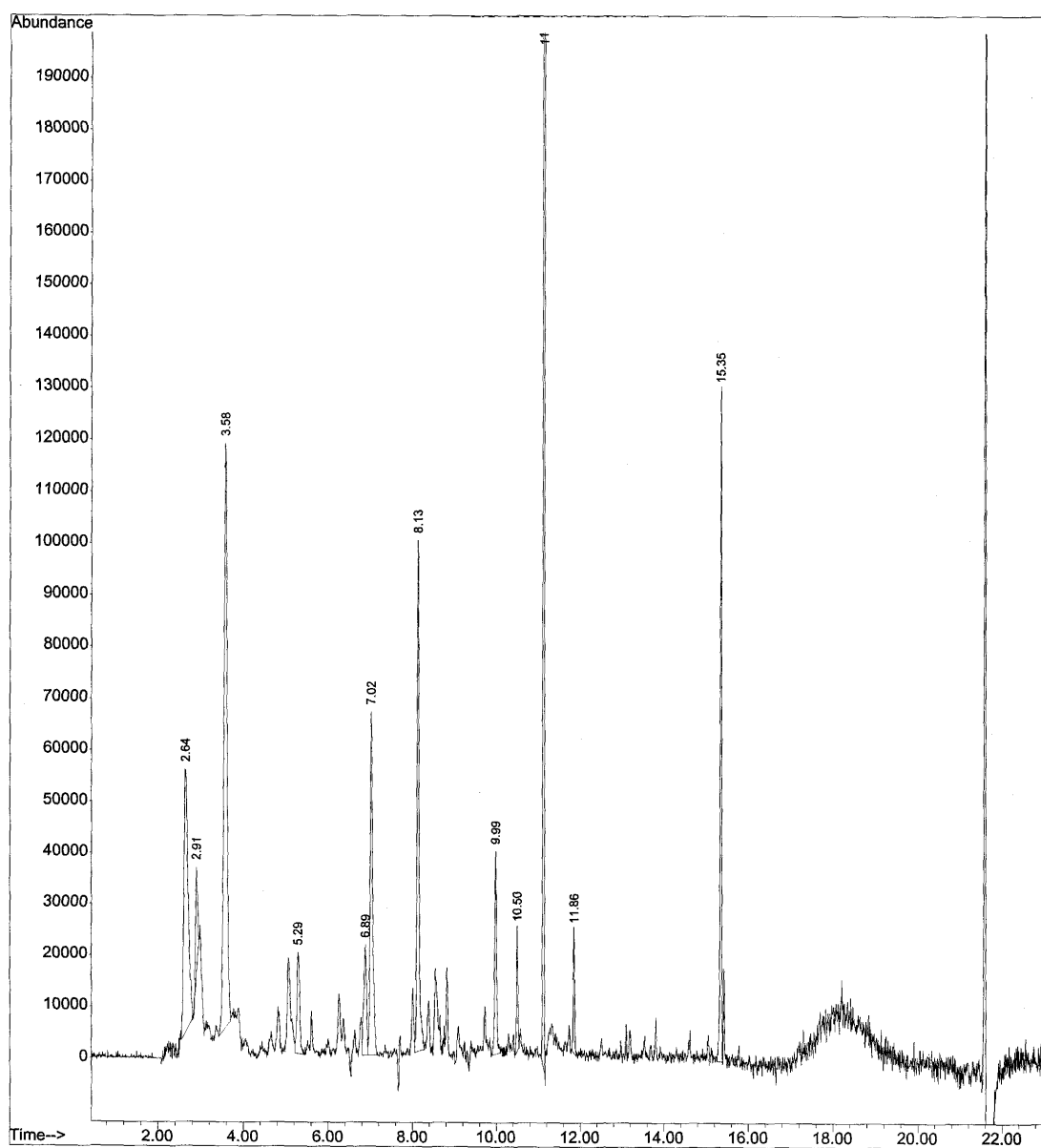


Figure 10. Background subtracted GC/MS chromatogram taken at 5 minute interval.

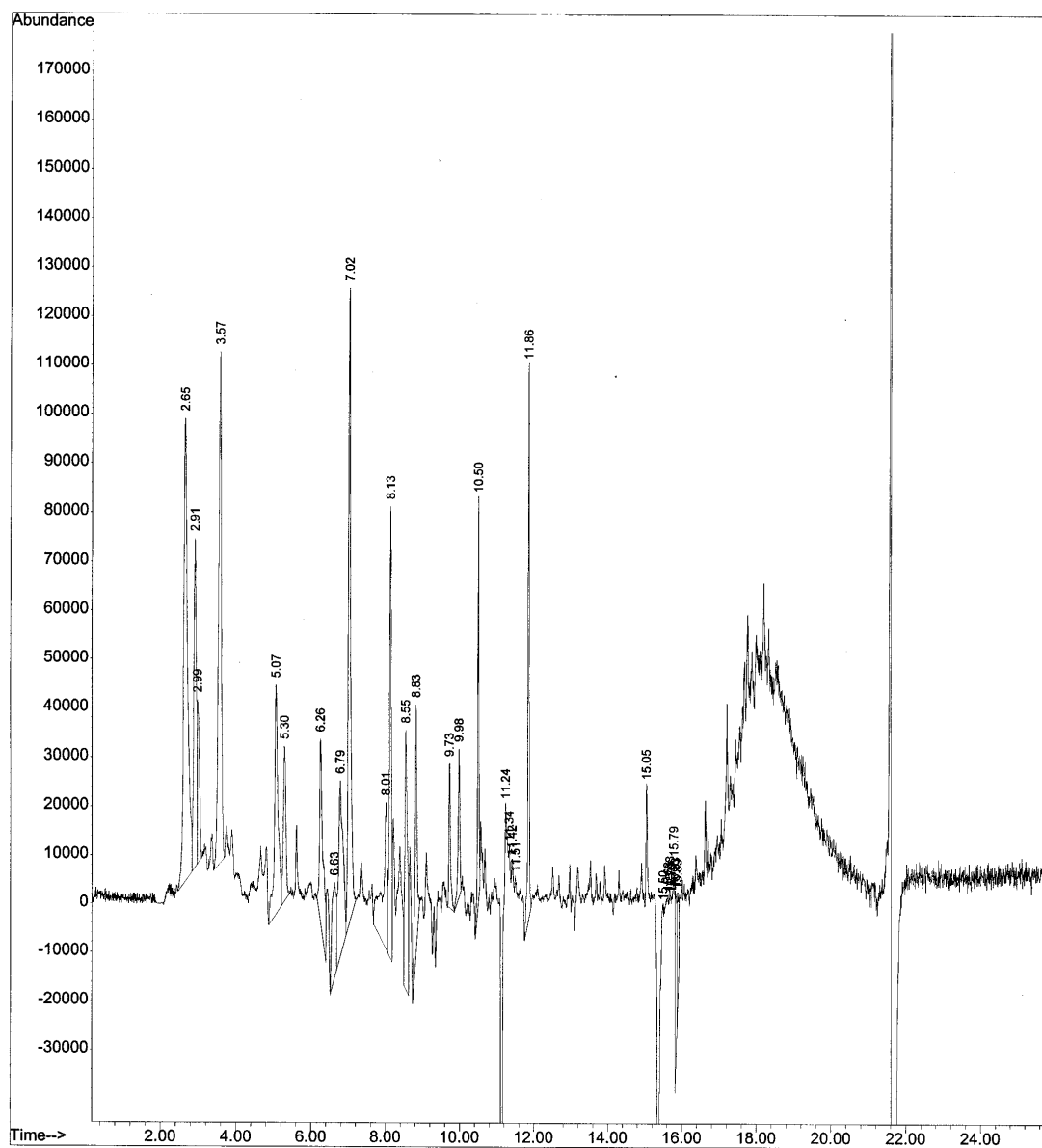


Figure 11. Background subtracted GC/MS chromatogram taken at 15 minute interval.

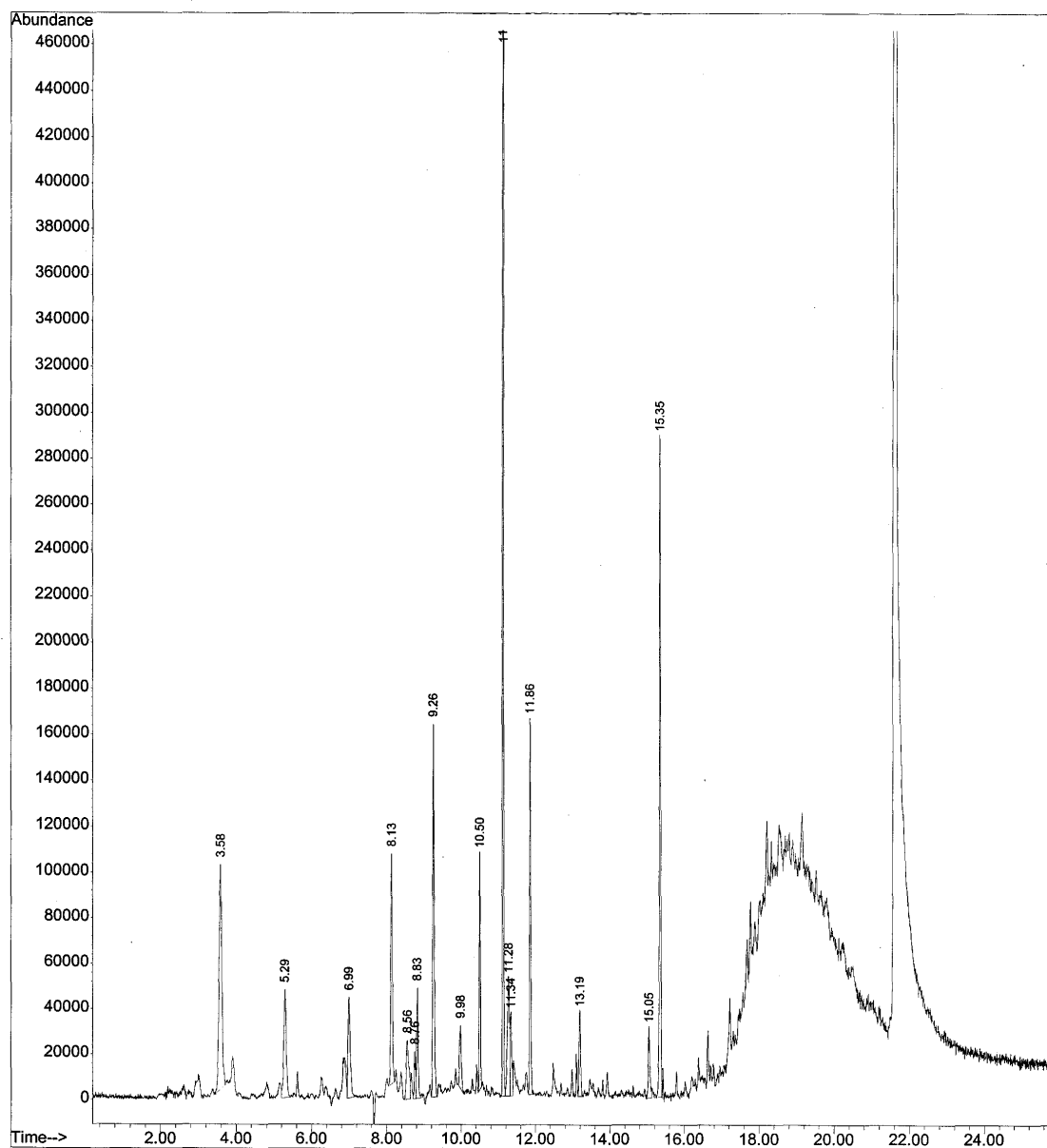


Figure 12. Background subtracted GC/MS chromatogram taken at 25 minute interval.

TABLE 3. VOC's detected by thermal desorption GC/MS.

<u>Retention time (min)^a</u>	<u>Compound^b (TLV-TWA in ppm)</u>
2.67	acetone (500 ppm)(CAS # 67-64-1)
2.96	butanal (CAS # 123-72-8)
3.63	benzene (0.5 ppm) (CAS # 1076-43-3)
5.36	hexanal (CAS # 66-25-1)
6.31	hexanone (5 ppm)(CAS #589-38-8)
7.08	acrylamide (CAS # 79-06-1)
8.20	benzaldehyde(CAS # 100-52-7)
9.26	hexanol(CAS # 111-27-3)
10.50	nonanal(CAS # 124-19-6)
11.86	decanal(CAS # 112-31-2)

^aRetention times are not meant to be exact. Slight differences do occur between samples.

^bCompounds with trace abundances much less than presented compounds are not listed. Compounds resulting from column bleed are also not listed. TLV-TWA's are shown for those compounds listed by the American Conference of Governmental Industrial Hygienists (ACGIH).⁸

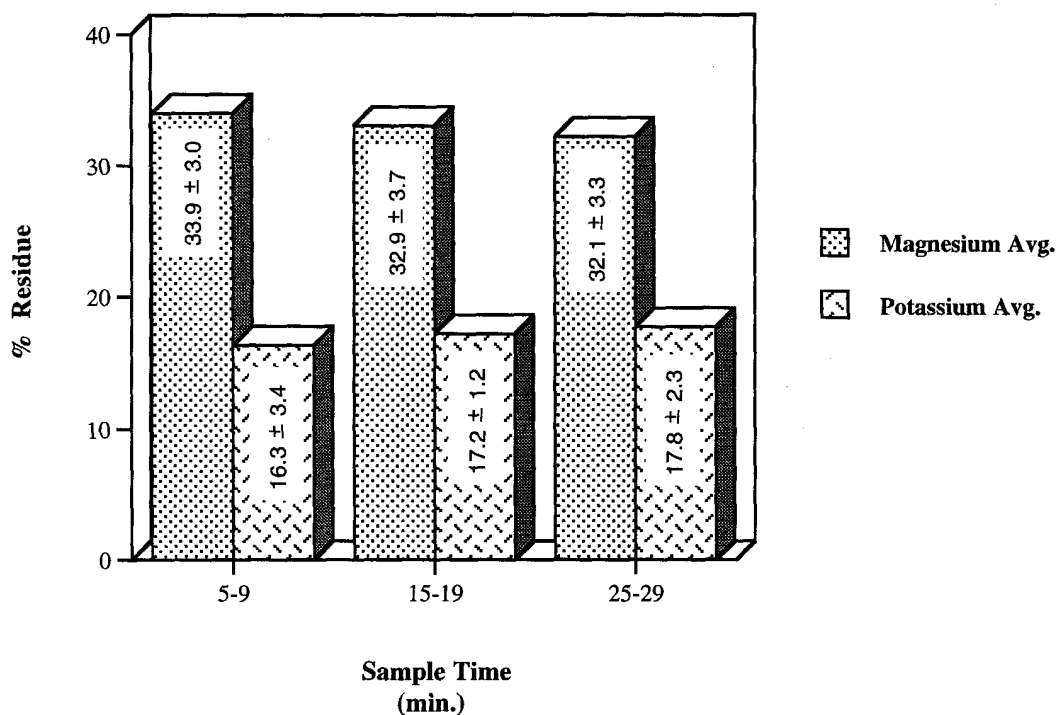


Figure 13. Mean percentages of elemental magnesium and potassium compounds in solid residue (principally carbonates and hydroxides).

ACKNOWLEDGEMENTS

The authors would like to thank Joe Domanico and Gene Tracy, Engineering Directorate, for their help in receipt and storage of the simulators. Thanks are also in order to Robyn Lee, Robyn B. Lee and Associates, for her helping with the statistical analysis.

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APPENDIX –INSTRUMENT CONDITIONS

A. Particle size analysis

TSI Inc. Aerodynamic Particle Sizer

Model No.: 3310
 Photomultiplier tube background: 0.262
 Nozzle(Δ P): 3.041
 Sheath Flowmeter: 3.401
 Total Flowmeter: 3.910

Diluter

Model No: 3302
 Flowrate: 0.05 SLPM
 Aerosol path(Δ P): 0.34”
 Total path(Δ P): 0.60”

B. Thermal Desorption Gas Chromatograph/ Mass Spectrometer analysis

Thermal desorption conditions

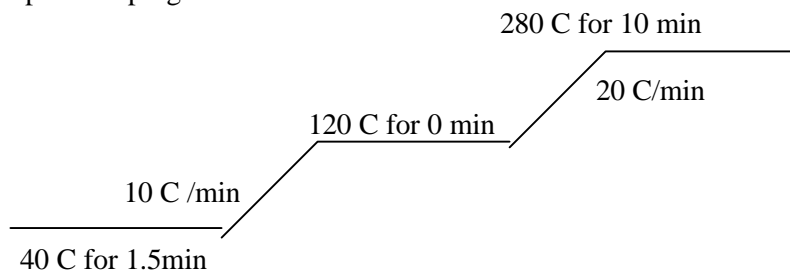
Instrument: Dynatherm ACEM900 Thermal Desorption Unit

Valve temp: 150 C	Dry time: 1 min
Tube temp: 250 C	Tube heat: 3 min
Transfer line temp: 200 C	Tube cool: 1 min
Trap temp: 250 C	Trap heat: 3 min

GC conditions

Instrument: HP 6890 Gas Chromatograph
 Column: HP-5MS 30m x 0.25mm x 0.25 μ m
 Column Flow(He): 1.0 ml/min (velocity 36 ml/min)(head pres 7.06 psi)

Temperature program



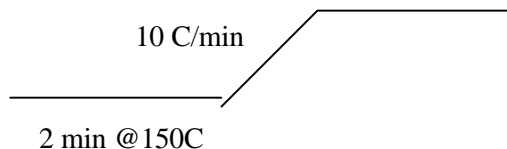
Detector conditions

Detector: HP5973 mass selective detector
 MS quad temp: 150 C
 MS source temp: 230 C
 Scan parameters: 30-550 amu ; threshold 150

C. GC-FID analysis

Instrument: HP 5890 Gas Chromatograph
Column: J+W Scientific DB-5 30m x 0.53mm x 1.5um
Liner: Single Taper (HP part number 5181-3316)
Injection volume: 2 ul splitless
Column flow(He): 1.0 ml/min (velocity 36 ml/min)(head press = 8.5 psi)
Inlet purge: Off time: 0 min; On time: 0.5min
Injector temp: 220 C

Temperature program 15 min @300 C



Detector conditions

Detector: Flame ionization detector
Detector temp: 300 C
Detector flow: 400 ml/min (air); 30 ml/min (hydrogen)

D. Atomic Absorption analysis

Instrument: Perkin Elmer 2380 Atomic Absorption Spectrophotometer
Air Flow: 40 psi
Acetylene flow: 20 psi

Potassium analysis

$\lambda = 766.5$
slit width = 0.7
lamp operating current = 30

Magnesium analysis

$\lambda = 284.2$
slit width = 0.7
lamp operating current = 20